"SHEAR AND TIME DEPENDENT VISCOUS BEHAVIOR OF CERAMIC SUSPENSIONS AT LOW SHEARS IN A BROOKFIELD VISCOMETER"

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1. INTRODUCTION

Colloidal processing of advance ceramic has received an increasing amount of attention during past decade. Colloidal processing technique such as slip casting, tape casting, centrifugal casting, injection molding and dip coating are employed to produce advance ceramics of improved reliability [1-3]. In all types of suspensions shape forming technique of ceramic materials, rheological properties of concentrated suspension play a vital role in controlling the shape forming behavior and optimizing the properties of green body. The particle size, the size distribution and the surface area of the particle also play important role in rheological study [4-7]. In order to achieve high solids in ceramic suspension, processing parameters such as powder characterization, power dispersion and wetting, slurry rheology, shear rate, volume of suspension etc. require a careful consideration. A proper control on these parameters will produced a super green microstructure with good particles packing and minimal defects such as agglomerates and pores. Further, the manufacturing processes in which large volume of concentrated suspensions are handled such as the production of solid rocket propellants and of various filled polymeric materials requires the understanding and control of basic rheological behavior of concentrated suspension [8-10]. However, high solid loadings can be attained with a small increase in viscosity using suspension having a distribution of particle sizes rather than the particles of the similar sizes. Most investigation of rheology of suspension, however, concentrates on prediction and correlation the viscosities of suspensions containing the uni modal size distribution, i.e., mono dispersed suspensions [11-12].

To characterize the dispersions viscosity measurements are widely used for studying their fundamental properties as well as an assessment of their suitability for a particular technological purpose. Ceramic dispersion systems are generally known to exhibit non - linear viscosity behavior and can be both shear dependent such as shear thinning and shear thickening as well as time dependent such as thixotropy and shear rejuvenation [13-14]. An

understanding of the equilibrium and transport properties of these slurries is necessary for the successful exploitation of these materials.

Moreover, rheological measurements are widely used to characterize dispersions, both as a means of studying their fundamental properties and as assessment of their suitability for particular technological purposes. Knowledge of their rheological behavior is essential for almost all dispersion in order to handle them satisfactorily. A numerous methods such as control tests for degree of dispersion, particle size analysis, sedimentation behavior, rheological determination, and optical analysis are established for characterization of dispersion. Among them rheological determination method play a very significant role in the assessment of the dispersion [15].

The characterization of colloidal suspensions depends upon the purposes for which the information is sought, because the total description would be an enormous task. Among the properties to be considered are the nature, surface groups, size, shape defects, pores, adsorbed surface films, internal surface stress, stability, and state of agglomeration. The rheological behavior of the dispersion is of considerable importance in a large number of industries and indeed many of the popular terms used to describe consistency are derived from the properties of particular type of materials. Rheological properties have significant values in the production of dispersion where certain type of mill may operate satisfactorily only within a limited range of flow properties, but more particularly in the use of the finished products. The most important rheological measurement techniques to assess the state of aggregation in a suspension include steady state measurements, transient state measurements as well as dynamic (oscillatory techniques) measurements. Viscosity measurements are widely used to characterize dispersions both as a means of studying their fundamental properties as well as an assessment of their suitability for a particular technological purpose.

The particle motion in the suspension and flow behavior of solid suspension have also been found a great interest in the fields of industrial process and material development consisting high strength ceramics, rocket propellants and reinforced polymer composites. Industrial materials such as suspensions, physical gel, foam, wet granular materials or emulsions to form network of interacting element in given condition have a significant importance. It is also highly desirable to understand and correlate the effect of aging to their microstructure and physical properties such as surface area, pore volume and size.

2. RESEARCH OBJECTIVE

In an preliminary investigation, of a conventional well dispersed alumina ceramic suspension of moderate loading (55% w/w and 21% v/v) it was curiously observed that with continual shearing at low shears, the viscosity of the suspension showed gradual increase with shearing time. Such behavior of these convention ceramic suspensions to the best of our knowledge has never been reported earlier in the literature.

Usually, for ceramic suspensions the opposite behavior is observed where the viscosity decreases with increase in shearing time. Such an aspect is usually referred as "shear rejuvenation". Further, for poorly dispersed suspension, the behavior of the ceramic suspensions showed typical behavior of shear rejuvenation.

Thus, the objective of the present work is

- To find out a plausible explanation of the observed increase in viscosity of these well dispersed suspensions with time under continual low shears.
- To exploit these feature to see whether these phenomenon can be utilized for characterization of ceramic suspensions.
- To study the rheological behavior of the aged suspension and monitor its rheological behavior.
- Realizing that settling down of negatively buoyant particles under gravitation is also a shearing system, it would be interesting to explore whether such increase in viscosity of the suspension may stabilize the suspension rheologically.

3. LITERATURE REVIEW

Many workers [2, 3, 5, 8] have shown that the paint, ceramic, electronic and mechanical industries utilize well dispersed suspensions of particles in liquid as stock materials for the production of various end products, such as tape casting slurries, paints, inks, electrical products, magnetic fluids, CMP fluids, investment casting slurries etc. The typical steps of the colloidal route to prepare a well dispersed slurry is prepared involve the control of the packing of the powders on a particulate scale. Viscosity measurements are widely used to characterize dispersions both as a means of studying their fundamental properties as well as an assessment of their suitability for a particular technological purpose. Ceramic dispersion

systems [16] are generally known to exhibit non - linear viscosity behaviour and can be both shear dependent such as shear thinning and shear thickening as well as time dependent such as thixotropy and shear rejuvenation. These behaviours are generally explained in terms of the changes in the size and structure of the particulates and depend upon the applied shear rate. An understanding of the equilibrium and transport properties of these slurries is necessary for the successful exploitation of these materials.

Rheopexy is also observed for layered clay materials which typically has a high aspect ratio in the range of 100-1500. The rheopexy in these systems is explained on the basis of the orientation of the tactoids normal to the direction of flow. It has been reported that below a critical shear rate especially in a Couette geometry, shear localization or shear banding may also result. Below this critical shear, it has been observed with the help of MRI–rheometer, all the flow is localized in a region close to shearing wall. It is also known that concentrated non aggregating mono disperse hard sphere suspension shear thickens and the shear thickening is a function of both phase volume and concentration in proportion to some maximum packing fraction which is itself partly controlled by the form of the particle size distribution. Several groups of workers [17-21] have demonstrated that shear thickening in concentrated colloidal suspension is due to the formation of jamming clusters bound together by hydrodynamic lubrication forces, often denoted by the term " hydroclusters ". For concentrated dispersions of monodisperse particles, a discontinuous jump in the viscosity has been observed [22-25]. It was also shown that stochastic processes such as Brownian motion destroys cluster formation resulting in reversible shear thickening.

Shear induced hydrodynamic diffusion [30-36] has been reported by various groups of particles migrating under the action of shear even under the conditions of sufficiently low Reynolds number (10^{-4}) where inertial forces are insignificant and a high Pė. A high Pė ensures negligible Brownian forces. Based on the pioneering observation of particle migration in a couette devise, and modelling of shear induced migration as a one dimensional diffusion process by Leighton and Acrivos (1987), shear induced diffusion have been studied quite extensively by both theoretically and experimentally under different conditions such as constant shear stress, constant shear rate, in narrow gap couette device, wide gap couette device, parallel plate geometry pressure driven channel flow, under steady state and also under dynamic conditions. All these studies involved mainly model neutrally buoyant, spherical and non colloidal particles.

In all these cases resultant particle flux(es) was observed because of existing anisotropy of stress or viscosity or streamline curvatures within the sheared suspension. The migration of particles usually took place from higher shear stress region to regions of lower stress or from higher viscous region to lower viscous region or from higher streamline curvature to lower streamline curvature of the suspension. Furthermore, it was also shown, in most of the cases, under appropriate conditions, the bigger the particles and higher the hydrodynamic forces applied, the greater was the migration of particles. There are also existing literature involving the migration of previously well settled negatively buoyant particles in the upward direction defying the forces of gravity under the conditions of very low Reynolds number and high Pė. This process of uplifting negatively buoyant particles is termed as viscous resuspension which may be significant in rheological suspensions to be characterized [34-35].

4. PROPOSED WORK

Present work will be carried out in following phase:

a) **First Phase**

b) **Second phase**

a) First Phase: Non aqueous medium

- Formulation of well dispersed alumina suspension in non aqueous medium:
- Incorporating controlled aggregation in the previously well dispersed suspensions bu using different concentrations of a suitable dispersant.
- Formulation of coagulated suspension with similar formulation but with non optimum dispersant concentration.
- Effect of loading on the phenomenon of positive drifts in viscosity.
- Effect of milling time on the phenomenon of positive drifts in viscosity.
- Formulation of similar well dispersed alumina suspension but having a different powder dimension.
- Characterization of all the ceramic suspensions by sedimentation tests, gloss measurements, microscopic studies, ESA measurements
- Variation of viscosity of the suspensions under continual long term constant shear.
- Variation of viscosity of the suspensions under changing shear.

- Response of the suspension to short term shear.
- Effect of aging the suspensions and observing the change in viscosity with time of the aged suspensions.
- Effect of the change in powder dimension, formulating well dispersed suspensions and its effect on the positive viscosity drifts.
- Taking all the results obtained from the above measurements and identifying a plausible explanation of such positive drifts of the well dispersed suspension.
- To understand if there exists any correlation between particle dimension (degree of aggregation) on the quantum of positive drifts.
- Finally, based on the above observations and the plausible explanations to elucidate the possibility of increasing the shelf life of well dispersed suspension rheologically by exploiting the positive drifts in viscosity under shear.

b) Second Phase : Aqueous Alumina suspension

- Special emphasis in this part will be given by changing the degree of aggregation of these aqueous suspension by changing the pH and monitoring the same through detailed ESA studies.
- All the rheological studies mentioned in the first phase will be carried out also for the aqueous suspension and will be used to both strengthen and verify the arguments deduced from the first phase of results.

5. METHODOLOGY

a) Materials used

Alumina powder A17NE and A16SG (ACC-Almatis), will be used for the present study. Characterization and composition of the powders is given in the following tables (table I and table II).

The binder used was Poly vinyl Butyral (PVB) of grade Maripol B-30 (Parekh Chemicals). The plasticizers used were Butyl Benzyl Phthalate [2- $(CH_3(CH_2)_3O_2C)C_6H_4CO_2CH_2\ C_6H_5]$ BBP,(Aldrich) and Poly Ethylene Glycol 400 $[HO(C_2H_4O)_nH$ where n = 380-400]; PEG, (Merck) .The solvent used was an azeotropic mixture of 50% (by weight) Methyl Ethyl Ketone ; MEK (Merck) and 50 % (by weight) of 99.9 % Ethanol; EtOH, (Changshu

Yangyuan Chemicals, China). Each of the individual solvents had a moisture content of 0.1% and was used as supplied.

Table I

Characteristics of A17NE and A16SG alumina powders taken for study

Alumina powder		***A 17 NE***	***A 16 SG***
Particle size		$d_{50} = 2.3\ \mu m$ $d_{90} = 7\ \mu m$	$d_{50} = 0.5\ \mu m$ $d_{90} = 2.0\ \mu m$
Specific Surface area/BET (m^2/g)		2.9	8.9
Chemical Composition	Al_2O_3	99.81%,	99.8%
	Na_2O	0.10%,	0.07%
	Fe_2O_3	0.03%,	0.02%
	MgO	0.01%,	0.05%
	SiO_2	0.03%	0.03%
	CaO	0.02%	0.02%

Table II

Composition of the Alumina Slurry and Formulation particulars

Components	**Function**	**Wt. % (vol. %)**
Polyethylene Glycol 400 (PEG) Merck, $[(C_2H_4O)_nH$ where n = 380-400]	Plasticizer	6.08(8.29)
Butyl Benzyl Phthalate (BBP) Aldrich, $[2\text{-}(CH_3(CH_2)_3O_2C)C_6H_4CO_2CH_2\ C_6H_5]$	Plasticizer	1.38(1.90)
Mixture of ethanol & Methyl ketone	Solvent	30.12(58.09)
Alumina	Powder	55.29(21.77)
Poly vinyl Butyral (PVB)	Binder	7.13 (9.95)

b) Preparation of slurries

The slurry (Table I) was prepared by vacuum heating the powders at 100^0 C for 7days prior to their use. Immediately after use the powder was once again kept under vacuum conditions. This ensures a negligible moisture content of the powders (Mistler and Twiname 2000) [21]. In the first stage, to a portion (13.96%) of the total azeotropic solvent mixture utilized in the formulation , 6.07% by weight of PEG, 1.38% by wt BBP and 1.38% of the binder PVB was added .The resultant mixture was stirred and kept overnight. After the time period, 55.2% by wt of the heat treated powder was then weighed and poured into the above mixture containing solvent & plasticizers. The resultant slurry was subsequently ball milled at 73rpm for 10 h with 10mm alumina balls (138% by wt of the total formulation) as the grinding medium. In the second stage, the solution of 5.74 % by weight of (PVB) and 16.28% of solvent mixture (viscosity: 665 $\mp$ 5 mPa.s) was added to the slurry. The slurry was further ball milled for 14h. The weight of the container containing the slurry was measured before and after the ball milling process to rule out any solvent or material loss. The alumina suspension thus formed was poured into a container and was de-aired under slow stirring and in vacuum conditions (300 mm Hg) till no more bubbles were seen to form. The weight loss during the de-airing process was found to be nominal (0.2%). Since the solvents had negligible moisture content and the powders heated prior to their use the overall moisture content can be safely assumed to be negligible.

Graduated cylinders (Borosil) fitted with stoppers and sealed with Parafilm "M" (Pechiney Plastic Pakaging) were used to investigate the sediment nature. The graduated cylinders containing the slurry were placed in an oven fixed at 60^0 C for 72 h before the sediment nature was investigated.

c) Instruments used, techniques and methods

The supernatant of the suspension was obtained by centrifuging (Sigma 1-14) 10 x g for 10 min in a and decanting the upper portion of the suspension. In order to find out the loading of the supernatant, density of a large number of suspension with various loadings (wt/wt) e.g. 25%, 30%, 40% ,45%, 50%, 55% and 60%, were measured. A graph was plotted of the densities of the suspensions vis-a- vis the powder loading. The density of the supernatant was then interpolated to find out the corresponding loading of the supernatant.

Viscosity measurements were carried out with a programmable Brookfield concentric cylinder Rheometer DV-III ULTRA coupled with a small sample adaptor (SSA 13RP, Brookfield) with EZ-lock assembly and embedded RTD temperature probes. The spindle used was SC4-31. The specifications of the container and the spindle are given in Table II.

The data was gathered with the help of Rheocal program supplied by Brookfield at a time interval of 1 min. The temperature of measurements was usually kept constant at 20^0C ∓ 0.2 and in some cases 0^0C with the help of a Brookfield water bath TC 502. Equal volume of slurry (13.4 ml) was used for all the viscosity measurements. Prior to measurements, the slurries were kept at rest in the SSA till the required temperature was obtained. After the required temperature was reached, the suspensions were pre sheared ($\dot{\gamma}t$ = 1800) and the suspensions were again kept at rest for 30 min before shearing at a specific shear was started. The solvent evaporation during the course of the experiment was monitored by measuring the weight of the SSA with the slurry, prior to viscosity measurements and after the viscosity measurements. It was observed that a negligible amount of solvent loss took place (0.23% per hr) during the course of experiments. Following the detailed methodology of suspension preparation and sample preparation as outlined above, the viscosity curves of the suspensions of different batches were found to be reproducible and had a deviation of around ± 2% during multiple reproducibility checks.

For measuring the time dependent rheological behaviour of the suspension an extended shear up ramp was performed by sequentially shearing the suspension at 1 sec^{-1}, 4sec^{-1} and 8 sec^{-1} respectively, followed by a rest period of 30 min and again at 1 sec^{-1}. This was done after the suspension reached the desired temperature and the usual pre shear of 1800.The time of rotation at a specific shear rate was arbitrarily chosen as 60 min before a transition to the next shear rate. Similar condition of viscosity measurement was also carried with the supernatant of the slurry and for the viscosity measurements at 0^0 C. Similarly, a shear down ramp was also applied to the suspension by sequentially shearing the suspension at 8 sec^{-1}, 4sec^{-1} and 1 sec^{-1} respectively, followed by a rest period of 30 min and again at 8 sec^{-1} respectively.

The supernatant of the suspension was obtained by centrifuging the suspension at 10 x g for 15 min with a Sigma 1-14 centrifuge. The supernatant thus formed was carefully decanted and was used for viscosity measurements. In order to find out the loading of the supernatant, density of a large number of suspension with various loadings (wt/wt) e.g. 10 %, 20 % ,30%, 40% , 50%, 55% and 60%, were measured. A graph was plotted of the densities of the suspensions vis-a- vis the powder loading. The density of the supernatant was then intrapolated to find out the corresponding loading of the supernatant.

The microscopic studies of the unprocessed powder and ball milled slurry as per formulation were performed with a metallurgical optical microscope fitted with a digital camera at a magnification of 500 x. Pseudoplasticity of the suspension was monitored by

measuring the viscosity of suspension at different shear rates by rotating the spindle for 10 rotations before a reading was recorded and the transition to a higher shear rate. The viscometer was programmed at a slow shear rate of 1 sec^{-1} and subsequently the viscometer speed was increased step wise to higher speeds (shear up) of 4 sec^{-1}, 6 sec^{-1}, 8 sec^{-1},10 sec^{-1}, 12 sec^{-1} and 14 sec^{-1} respectively.

6. WORK ALREADY DONE (RESULTS OBTAINED)

Major results are shown in the followings figures:

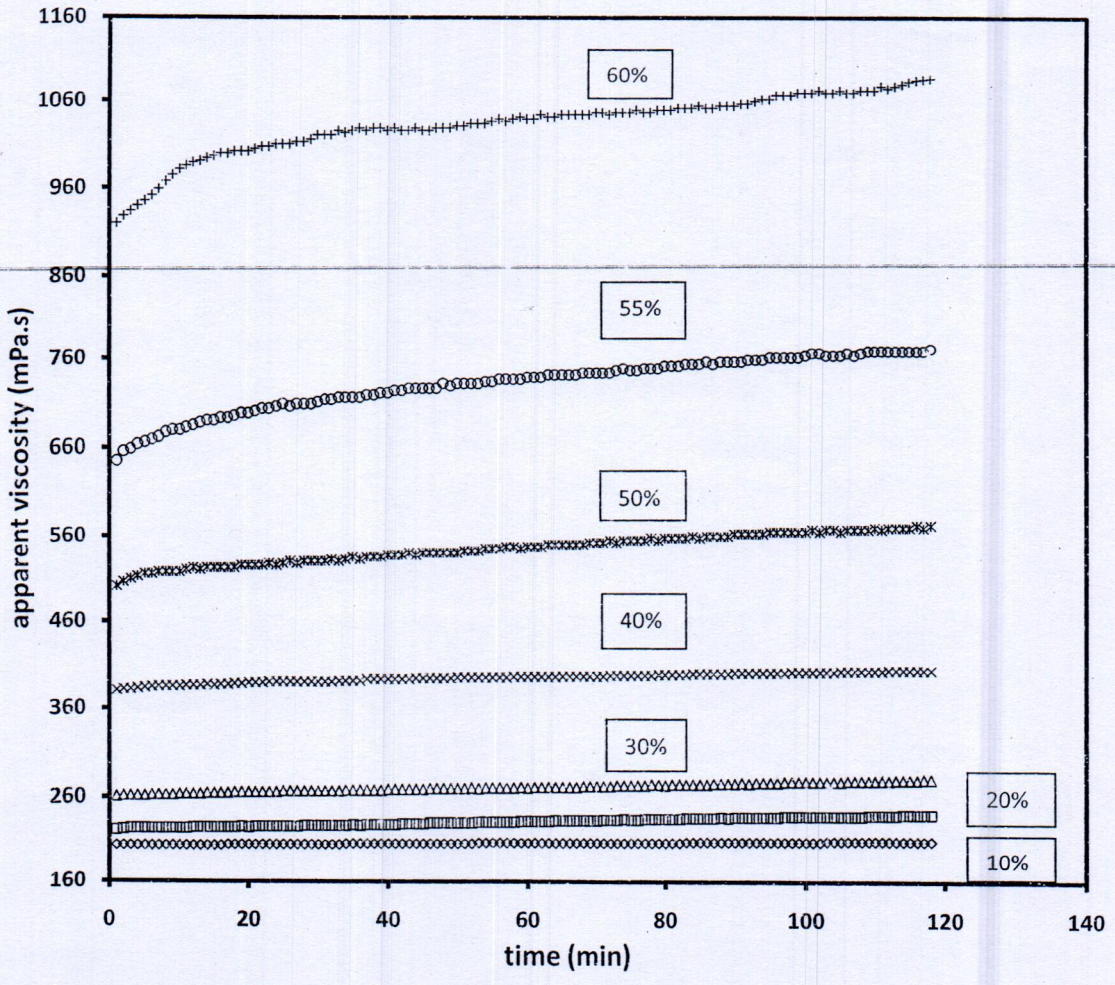

Fig1. Effect of powder loading

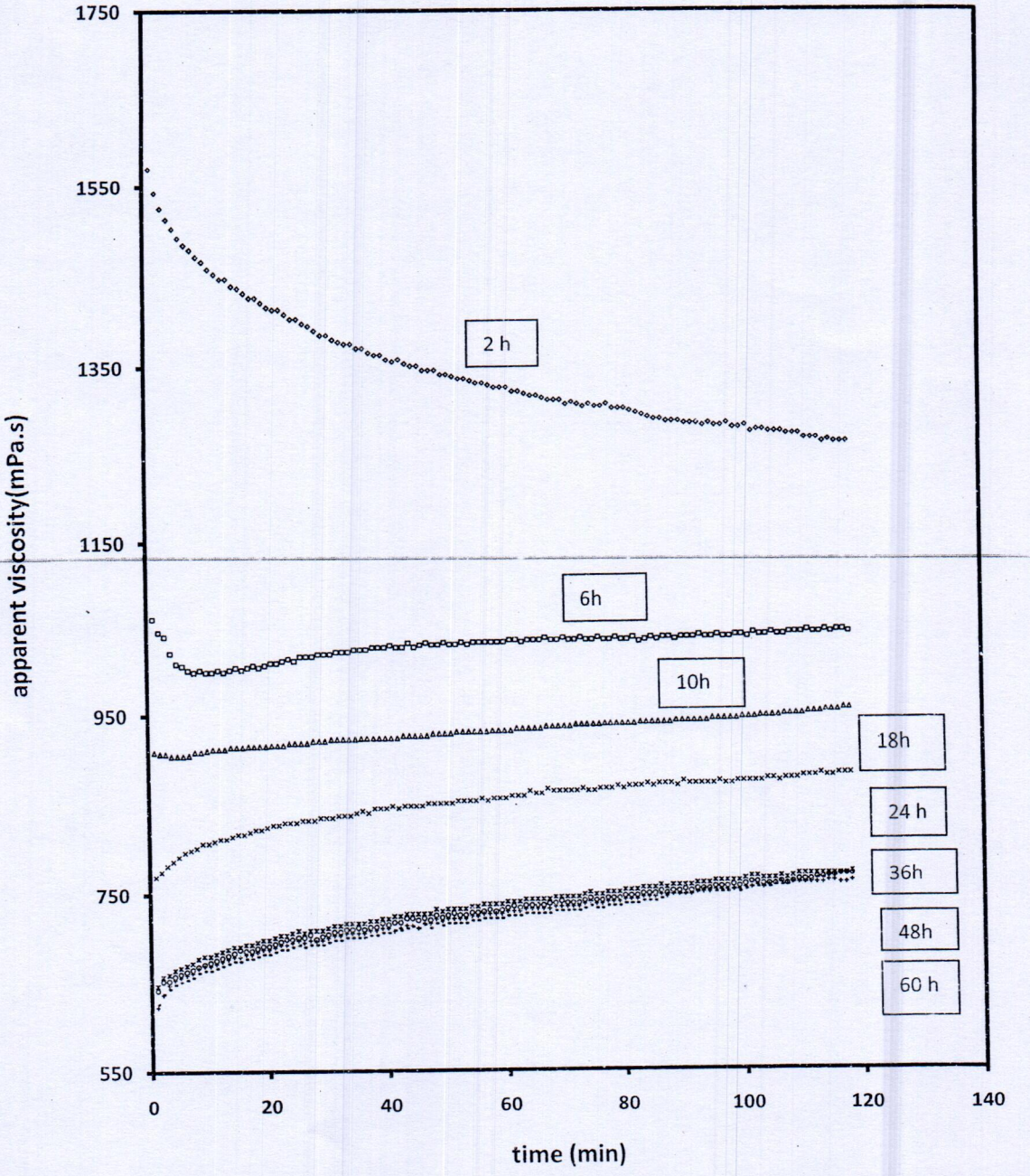

Fig2. Effect of milling time

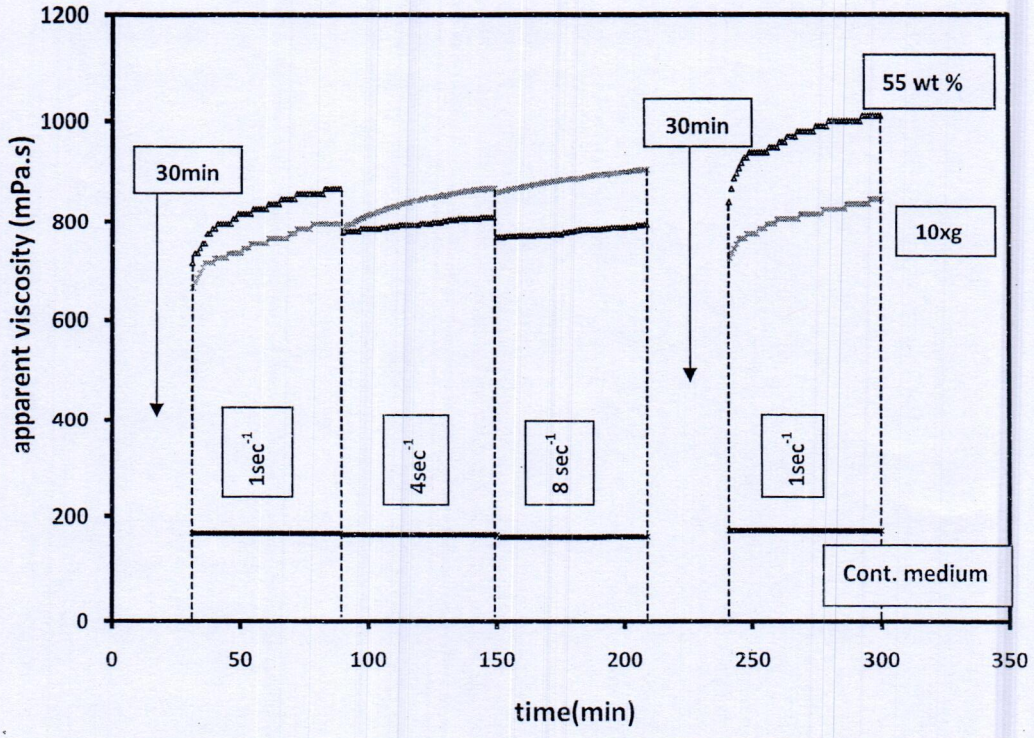

Fig3. Shear Up Ramp showing flow behavior of supernatant and mother slurry

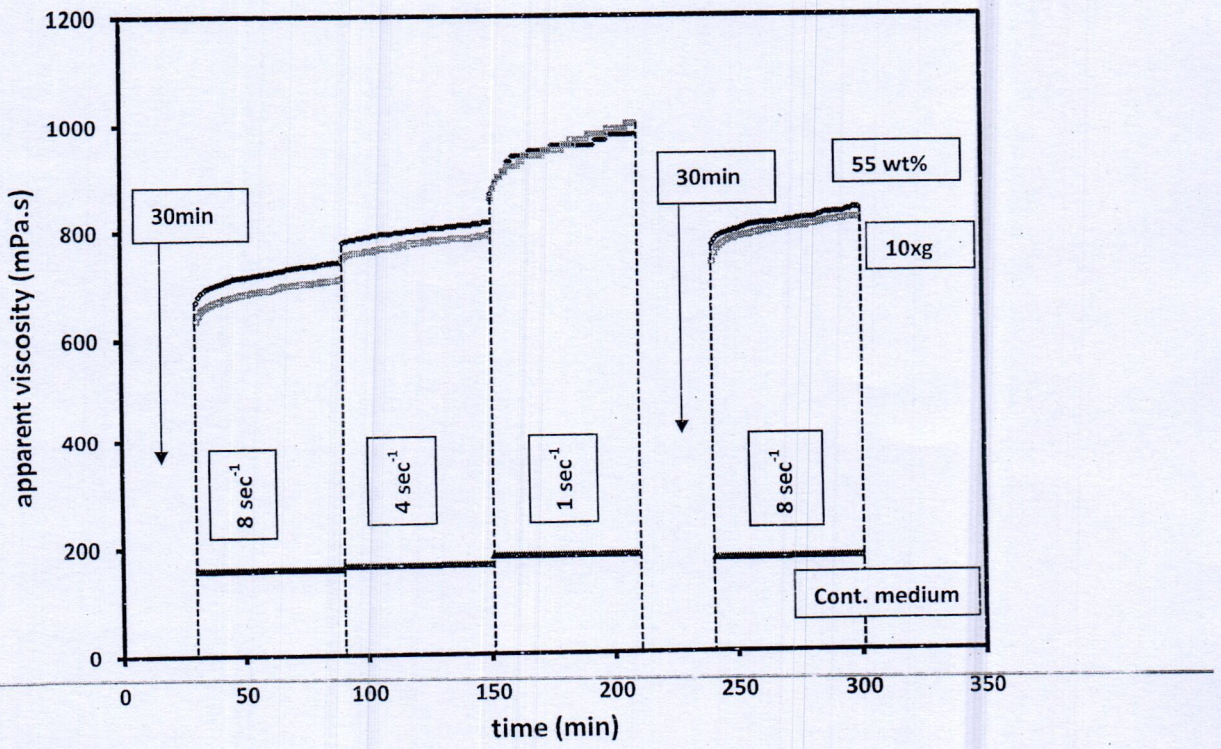

Fig4. Shear down Ramp showing flow behavior of supernatant and mother slurry

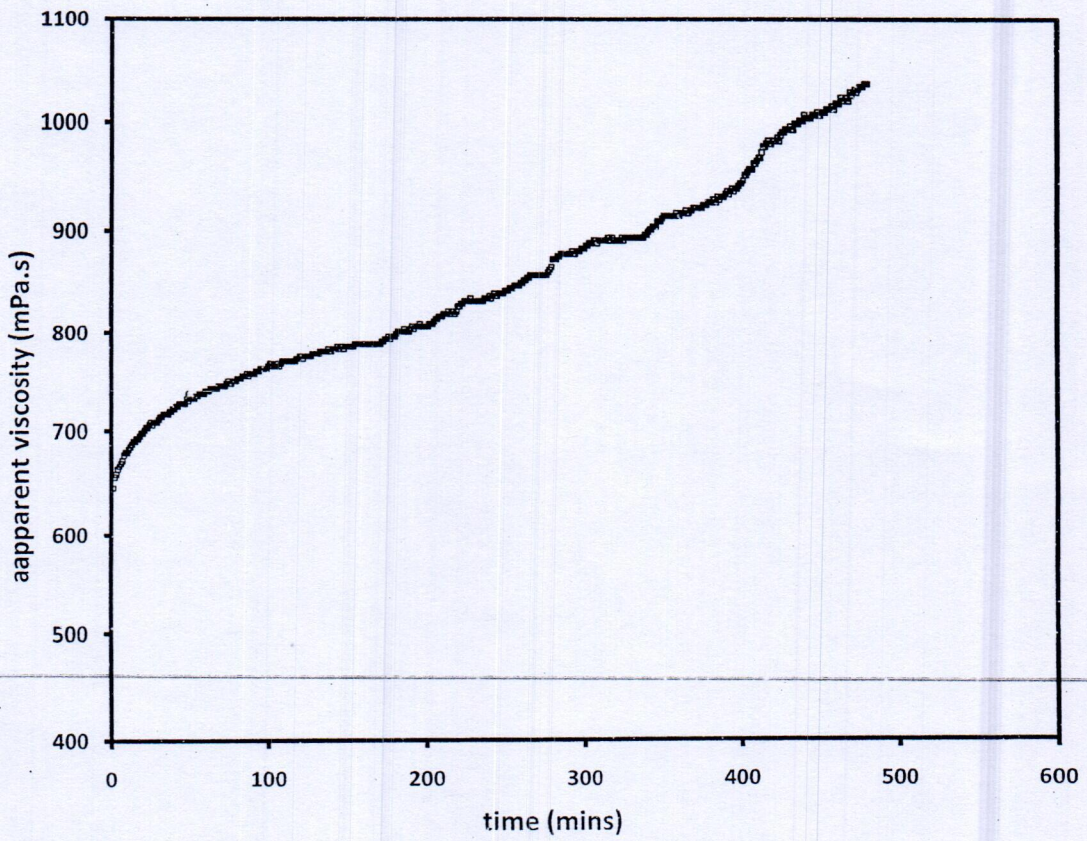

Fig5. Apparent viscosity behavior with time

7. DISCUSSION

a) Thus, we report here transient shear and time dependent behavior of well dispersed alumina suspension having different degree of aggregation stabilized by Phosphate Ester at low shear transient domain. This identification represents the possibility of numerous effects such as state of agglomeration, [8, 9, 10] internal surface stress, shear applied, and degree of shear induced diffusion which may alter the stability of suspensions. Results of this investigation are very essential for the successful exploitation of materials to understand the equilibrium and transport properties of the suspension.

b) The present investigation conducted with 55% (wt/wt) alumina suspension having different degree of aggregation measures the transient viscosity which shows diverse

time dependent and shear dependent viscosity characteristics in the low shear domain. The corresponding drift in viscosity for these suspensions will be different under similar shearing conditions. However, to the best of our knowledge a time and shear dependent behaviors of suspensions at low shear rates are hitherto unreported in literature. It will be subsequently shown in this report that such investigation reflects some fundamental physics occurring in the suspension under flow which may also open up a new vista for characterizing well dispersed suspensions.

c) A comparative analysis of shear and time dependent behavior of well dispersed alumina suspension of A17NE alumina and A16SG alumina. These out comings give a platform to establish a relation between model system and real system for the description of new approaches in suspension rheology.

8. PAPER PUBLICATIONS IN JOURNALS/CONFRENCES

Sr. No	Authors	Title of paper	Name of the Journal	Volume	Pages	Year
1.	A.Mukherjee, D.Sharma, H.L.Singh, S.C.Chauhan	**The Time dependent and Shear dependent Transient Viscosity of an Alumina suspension**	Mechanics of time Dependent Materials	Re-communicated		
2.	A.Mukherjee, D.Sharma	**Transient viscosities of concentrated well dispersed Al2O3 Suspensions**	Mechanics of time Dependent Materials	Under communication stage		
3.	A.Mukherjee, D.Sharma, H.L.Singh, S.C.Chauhan	**Transient Viscosity of negatively buoyant suspensions of Alumina.**	Mechanics of time Dependent Materials	Under communication stage		
4.	A.Mukherjee, D.Sharma,	**Comparative Study of Transient Viscosity of Negatively Buoyant Suspensions with varying particle sizes.**	Mechanics of time Dependent Materials	Under communication stage		

9. CONCLUSIONS

- The present investigation conducted with 55% (wt/wt) alumina suspension measures the transient viscosity which shows diverse time dependent and shear dependent viscosity characteristics in the shear range 1 sec^{-1} – 8 sec^{-1}. The viscosity of the suspensions was found to increase at each of the shear rates employed.

- The degree of viscosity increase was found to be substantial and a function of both powder loading and milling time respectively.
- Further, this investigation shows that the overall observed viscosity curve is the resultant of two major competitive processes of shear induced hydrodynamic diffusion and shear rejuvenation.
- The results obtained indicated than the drifts in viscosity of the well dispersed suspensions can be a basis for identifying the best suspension in a series of well dispersed suspensions.
- The present investigation shows strong indication that the physics behind shear induced diffusion may be utilized to characterize and identify the best dispersion in a series of finely balanced negatively buoyant particulate suspensions by studying the time dependent viscosity of these suspensions.

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